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(21) International Application Number: PCT/US97/13015 (22) International Filing Date: 24 July 1997 (24.07.97) (30) Priority Data: 08/698,011 13 August 1996 (13.08.96) US (71) Applicant: CHEVRON CHEMICAL COMPANY [US/US]; 555 Market Street, San Francisco, CA 94105 (US). (72) Inventors: KATSUMOTO, Kiyoshi; 2615 Brooks Avenue, El Cerrito, CA 94530 (US). CHING, Ta, Yen; 10 Santa Yorma Court, Novato, CA 94945 (US). (74) Agents: MICHEL, Marianne, H. et al.; Chevron Corporation, Law Dept., P.O. Box 7141, San Francisco, CA 94120-7141 (US).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: MULTI-COMPONENT OXYGEN SCAVENGING COMPOSITION (57) Abstract An oxygen scavenging composition or system is provided comprising at least one polyterpene and at least one catalyst effective in catalyzing an oxygen scavenging reaction. A film, a multi-phase composition, a multi-layer composition, an article comprising the oxygen scavenging composition, a method for preparing the oxygen scavenging composition, and a method for scavenging oxygen are also provided.		

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1 **MULTI-COMPONENT OXYGEN SCAVENGING COMPOSITION**

2 This application is a continuation-in-part of co-pending application Serial
3 No. 08/388,815 filed February 15, 1995.

4 Background of the Invention

5 The present invention relates to an oxygen scavenging composition or system
6 which can be employed in films, multi-layer films, sheets and molded or
7 thermoformed shapes that find utility in low oxygen packaging for
8 pharmaceuticals, cosmetics, oxygen sensitive chemicals, electronic devices, and
9 food.

10 Organic oxygen scavenging materials have been developed partly in response to
11 the food industry's goal of having longer shelf-life for packaged food.

12 One method which is currently being employed involves the use of "active
13 packaging" where the package is modified in some way so as to control the
14 exposure of the product to oxygen. Such "active packaging" can include sachets
15 containing iron based compositions such as AGELESSTM which scavenges
16 oxygen within the package through an oxidation reaction. However, such an
17 arrangement is not advantageous for a variety of reasons including the
18 accidental ingestion of the sachets or the oxygen-scavenging material present
19 therein.

20 Other techniques involve incorporating an oxygen scavenger into the package
21 structure itself. In such an arrangement, oxygen scavenging materials constitute
22 at least a portion of the package, and these materials remove oxygen from the
23 enclosed package volume which surrounds the product or which may leak into

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1 the package, thereby in the case of food products, inhibiting spoilage and
2 prolonging freshness.

3 Oxygen scavenging materials include low molecular-weight oligomers that are
4 typically incorporated into polymers or can be oxidizable organic polymers. Such
5 oxygen scavenging materials are typically employed with a suitable catalyst, e.g.,
6 an organic or inorganic salt of a transition metal catalyst such as cobalt
7 neodeconate, cobalt stearate, etc.

8 Often, these oxygen scavenging compositions are not effective at low
9 temperatures. The compositions require a long induction period or do not
10 scavenge oxygen under the storage environments for certain packaged food
11 applications.

12 Another major problem is that a wide variety of organic compounds are produced
13 upon oxidation of certain oxygen scavenging materials. Many of these oxidation
14 products can migrate from the oxygen scavenging material and enter the
15 headspace surrounding the food or even enter the food itself. Some oxidation
16 products, such as low molecular weight aldehydes and carboxylic acids, have
17 foul odors or unpleasant taste or can be compounds that are otherwise
18 undesirable.

19 Summary of the Invention

20 It is an object of the present invention to provide a composition effective for
21 oxygen scavenging.

22 It is another object of the present invention to provide a composition effective for
23 oxygen scavenging at low temperatures.

1 It is another object of the present invention to provide a composition which
2 produces reduced levels of oxidation by-products.

3 It is another object of the present invention to provide an article, package or
4 container suitable for oxygen scavenging.

5 It is another object of the present invention to provide a method for preparing an
6 oxygen scavenging composition.

7 It is another object of the present invention to provide a method for scavenging
8 oxygen.

9 According to the present invention, an oxygen scavenging composition or system
10 is provided comprising at least one polyterpene and at least one catalyst
11 effective in catalyzing the oxygen scavenging reaction. A film, a multi-phase
12 composition, a multi-layer composition, an article comprising the oxygen
13 scavenging composition, a method for preparing the oxygen scavenging
14 composition, and a method for scavenging oxygen are also provided.

15 Brief Description of the Drawings

16 Figure 1 graphically shows the oxygen scavenging performance of an oxygen
17 scavenging composition comprising 30% polyterpene and 70% polyethylene.

18 Figures 2-4 show by bar graphs the relative amounts of specific aldehydes
19 produced from examples containing blends of polyethylene with polyterpene,
20 styrene/butadiene block copolymer, polybutadiene, or polyoctenamer.

21 Figure 5 shows the relative amounts of specific acids produced from examples
22 containing blends of polyethylene with polyterpene, styrene/butadiene block
23 copolymer, polybutadiene, or polyoctenamer.

1 Figure 6 shows the relative amounts of specific alkenes produced from examples
2 containing blends of polyethylene with polyterpene, styrene/butadiene block
3 copolymer, polybutadiene, or polyoctenamer.

4 Detailed Description of the Invention

5 It has been found that polyterpenes are especially effective oxygen scavenging
6 materials particularly at low temperatures, e.g., refrigerated food temperatures.
7 Examples of such compounds include poly(alpha-pinene), poly(dipentene),
8 poly(beta-pinene), poly(d-limonene), and poly(d,l-limonene).

9 The polyterpenes can be introduced into the oxygen scavenging system by a
10 variety of techniques. The polyterpenes can be formed into films, coated onto a
11 material such as aluminum foil or paper, formed into bottles or other rigid
12 containers, or even incorporated into a material such as paper, for example, in
13 flexible and rigid packaging. The polyterpene can also be in a localized area on
14 a layer, for example, it may be in a patch that is laminated to another layer.

15 The polyterpene is generally present in an amount sufficient to scavenge at least
16 0.1 cc O₂/gram of oxygen scavenging composition/day. Preferably, it is capable
17 of scavenging at least about 0.5, and more preferably at least about 1 cc
18 O₂/gram of oxygen scavenging composition/day.

19 The amount of polyterpene employed in the oxygen scavenging composition can
20 vary broadly depending on the desired characteristics of the final product.
21 Generally, the polyterpene is present in an amount in the range of from about
22 5 weight percent to about 95 weight percent based on the total oxygen
23 scavenging composition, preferably from about 10 weight percent to about
24 75 weight percent, and more preferably from 15 weight percent to 50 weight
25 percent.

1 The polyterpene can be blended with a carrier resin comprising other oxidizable
2 polymers or polymers having a slower oxidation rate than the polyterpene.

3 Examples of other oxidizable polymers include substituted or unsubstituted
4 ethylenically unsaturated hydrocarbons such as polybutadiene, polyisoprene,
5 and styrene-butadiene block copolymers. Other examples include those
6 described in U.S. Pat. Nos. 5,211,875 and 5,346,644 to Speer et al., which are
7 hereby incorporated by reference in their entirety. Other examples include
8 poly(meta-xylenediamine-adipic acid) (also known as MXD6), acrylates which
9 can be prepared by transesterification of poly(ethylene-methyl acrylate) such as
10 poly(ethylene-methyl acrylate-benzyl acrylate), poly(ethylene-methyl acrylate-
11 tetrahydrofurfuryl acrylate), poly(ethylene-methyl acrylate-nopol acrylate) and
12 mixtures thereof. Such transesterification processes are disclosed in 08/475,918
13 filed June 7, 1995, the disclosure of which is hereby incorporated by reference.

14 In a preferred embodiment, the carrier resin oxidizes at a slower rate than the
15 polyterpene. Oxygen scavenging compositions prepared from such carrier
16 resins produce reduced amounts of migratory oxidation by-products such as low
17 molecular weight aldehydes, alkenes and carboxylic acids.

18 Typical examples of carrier resins exhibiting a slower oxidation rate include
19 polyesters, polyaromatics, or polyolefin homopolymers, copolymers, or
20 terpolymers. Specific examples of polymers exhibiting a slower oxidation rate
21 include polyethylene, low density polyethylene, high density polyethylene, linear
22 low density polyethylene, polystyrene, as well as copolymers such as
23 poly(ethylene-vinyl acetate), poly(ethylene-methyl acrylate), poly(ethylene-ethyl
24 acrylate), poly(ethylene-butyl acrylate), and ionomers of poly(ethylene-methyl
25 acrylate), poly(ethylene-ethyl acrylate), or poly(ethylene-acrylic acid).

- 1 Polyethylene including low density, linear low density, or ultra-low density
2 polyethylene is preferred due to its processability and versatility.
- 3 The amount of carrier resin employed can vary broadly. Generally, the carrier
4 resin is present in an amount in the range of from about 5 weight percent to
5 about 95 weight percent based on the total weight of the oxygen scavenging
6 composition, preferably from about 25 weight percent to about 90 weight
7 percent, and more preferably from 50 weight percent to 85 weight percent.
- 8 The catalyst can be any catalyst known in the art which is effective in initiating
9 the oxygen scavenging reaction. Typical catalysts include transition metal salts.
10 Suitable catalysts are disclosed in U.S. Pat. Nos. 5,211,875 and 5,346,644 to
11 Spear et al., the disclosures of which are hereby incorporated by reference in
12 their entirety. Cobalt compounds are preferred and cobalt oleate, cobalt
13 linoleate, cobalt neodecanoate, cobalt stearate and cobalt caprylate are
14 especially preferred.
- 15 The catalyst is present in an amount sufficient to catalyze the oxygen
16 scavenging reaction. Generally, the catalyst will be present in an amount in the
17 range of from about 50 ppm to about 10,000 ppm based on the total weight of
18 the oxygen scavenging composition, preferably from 100 ppm to 10,000 ppm,
19 and more preferably from 100 ppm to 5,000 ppm.
- 20 The catalyst can be introduced in any manner which does not react with and/or
21 deactivate the catalyst. For example, the catalyst can be applied onto the
22 oxygen scavenging material by any suitable means, e.g., coating techniques
23 such as spray coating, extrusion compounding (including masterbatching) or
24 lamination.

1 The oxygen scavenging composition can be activated by methods known in the
2 art such as ultraviolet, e-beam, or thermal triggering. Preferably, the composition
3 is activated with $0.2\text{--}5\text{ J/cm}^2$ of UV radiation in the range of from 250–400 nm. A
4 photoinitiator is useful for decreasing the catalyst activation time. Effective
5 photoinitiators include those known in the art.

6 In another aspect of the invention, the oxygen scavenging composition
7 comprises a first phase comprising the polyterpene and a second phase
8 comprising the catalyst. The first phase is essentially devoid of catalyst. The
9 second phase is in sufficiently close proximity to the first phase to catalyze the
10 oxygen scavenging reaction. When the polyterpene and the catalyst are in
11 separate phases, processing difficulties, such as deactivation of the catalyst, are
12 avoided.

13 In another aspect of the invention, the catalyst is incorporated into a polymeric
14 material to form at least one catalyst-containing layer. In such a case, the
15 catalyst-containing layer can be situated between the package contents and an
16 oxygen scavenging layer or between the outside of the package and the oxygen
17 scavenging layer. Also, the catalyst layer can be located between two oxygen
18 scavenging layers or the oxygen scavenging layer can be located between two
19 catalyst layers.

20 In another aspect of the invention, the oxygen scavenging composition or system
21 can include a polymeric selective barrier layer. The selective barrier layer
22 functions as a selective barrier to certain oxidation by-products, but not to
23 oxygen itself. Preferably, the layer prevents at least half of the number and/or
24 amount of oxidation by-products having a boiling point of at least 40°C from
25 passing through the polymeric selective barrier layer.

1 The oxygen scavenging composition can include additives, stabilizers,
2 plasticizers and UV sensitizers (i.e., photoinitiators) which do not interfere with
3 the oxygen scavenging function.

4 The oxygen scavenging compositions or systems can be employed in the
5 production of packages, both rigid and flexible, by techniques which are known
6 in the art.

7 The oxygen scavenging compositions of the present invention are especially
8 effective in low temperature environments. The compositions of the present
9 invention also produce reduced amounts of migratory oxidation by-products. Of
10 particular interest is the reduction of oxidation by-products such as low molecular
11 weight aldehydes, alkenes and carboxylic acids which can adversely affect
12 organoleptics.

13 The present invention is also useful in improving the shelf-life of packaged
14 oxygen-sensitive products such as pharmaceuticals, cosmetic, chemical,
15 electronic devices, health and beauty products. The system can also be used in
16 moldings, coatings, patches, bottle cap inserts and molded or thermoformed
17 shapes, such as bottles and trays. In all of these applications, the oxygen
18 scavenging composition effectively scavenges oxygen, whether it comes from
19 the headspace of the packaging, is entrained in the food or product, or originates
20 from outside the package.

21 The present invention will now be described further in terms of certain examples
22 which are solely illustrative in nature and should in no way limit the scope of the
23 present invention.

Examples

2 Blends of various resins were prepared as follows.

3 In Run 101, 350 g PE 1017 resin from Chevron (low density polyethylene) and
4 150 g Piccolyte C115 resin from Hercules (polylimonene) were melt blended at
5 170°C to give a blend of 70 weight percent polyethylene and 30 weight percent
6 Piccolyte. Figure 1 demonstrates the oxygen scavenging properties at 4°C of
7 the thus produced blend of Run 101. The percent oxygen in a closed 300 cc
8 headspace was measured on various days. The sample size was 0.25 g.

9 In Run 102, a blend of 90 weight percent Vector 8508D resin from
10 Dexco(styrene/butadiene block copolymer) and 10 weight percent PE 1017 was
11 prepared.

12 In Run 103, a blend of 54 weight percent Taktene 1202 rubber from Bayer
13 (polybutadiene) and 36 weight percent PE 1017 was prepared.

14 In Run 104, a blend of 30 weight percent Vestenamer resin from Huls
15 (polyoctenamer) and 70 weight percent PE 1017 was prepared.

The blends also contained 1000 ppm by weight Irganox 1076, and 1000 ppm by weight cobalt oleate. The blends were extruded into 1-1.5 mil thick films. The film samples were irradiated with a Blak-Ray UV lamp (254 nm, 5 mW/cm²) for 1 minute. Film samples were 1 inch away from the UV lamps. A predetermined amount of samples of the thus prepared films was individually placed in 2" x 30" glass tubes and purged at 20-25°C with 10-15 mL/min. one percent oxygen. The gas was trapped in 3 stages, trap 1 — ice bath, trap 2 — dry ice and acetone, and trap 3 — bubbled gas through water. The trapped gases from the samples were analyzed using gas chromatography and mass spectrometry.

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- 1 The relative amounts of specific by-products for Runs 101-104 are indicated by
- 2 the bars in Figures 2-6. White represents Run 101. Light gray represents
- 3 Run 102. Dark Gray represents Run 103. Black represents Run 104.

1 WHAT IS CLAIMED IS:

- 2 1. An oxygen scavenging composition comprising at least one polyterpene
3 and at least one catalyst effective in catalyzing oxygen scavenging.
- 4 2. The oxygen scavenging composition according to claim 1, wherein the
5 polyterpene comprises poly(alpha-pinene), poly(beta-pinene),
6 poly(dipentene), poly(d-limonene), or poly(d,l-limonene).
- 7 3. The oxygen scavenging composition according to claim 1 which exhibits
8 reduced amounts of oxidation by-products compared to substituted or
9 unsubstituted ethylenically unsaturated hydrocarbon polymers.
- 10 4. The oxygen scavenging composition according to claim 1 wherein the
11 polyterpene is present in an amount in the range of from about 5 weight
12 percent to about 95 weight percent based on the total oxygen scavenging
13 composition.
- 14 5. The oxygen scavenging composition according to claim 4 wherein the
15 polyterpene is present in an amount in the range of from about 10 weight
16 percent to about 75 weight percent based on the total oxygen scavenging
17 composition.
- 18 6. The oxygen scavenging composition according to claim 5 wherein the
19 polyterpene is present in an amount in the range of from 15 weight percent
20 to 50 weight percent based on the total oxygen scavenging composition.
- 21 7. The oxygen scavenging composition according to claim 1 further
22 comprising at least one carrier resin.

- 1 8. The oxygen scavenging composition according to claim 7 wherein the
2 carrier resin exhibits a slower oxidation rate than the polyterpene.
- 3 9. The oxygen scavenging composition according to claim 8, wherein the
4 carrier polymer is a polyester, a polyaromatic, or a polyolefin homopolymer,
5 copolymer, or terpolymer.
- 6 10. The oxygen scavenging composition according to claim 9, wherein the
7 carrier polymer is polyethylene, polystyrene, poly(ethylene-vinyl acetate),
8 poly(ethylene-methyl acrylate), poly(ethylene-ethyl acrylate), poly(ethylene-
9 butyl acrylate), or an ionomer of poly(ethylene-methyl acrylate),
10 poly(ethylene-ethyl acrylate), or poly(ethylene-acrylic acid).
- 11 11. The oxygen scavenging composition according to claim 10, wherein the
12 carrier polymer is polyethylene, poly(ethylene-methyl acrylate), or an
13 ionomer of poly(ethylene-methyl acrylate) or poly(ethylene-acrylic acid).
- 14 12. The oxygen scavenging composition according to claim 11, wherein the
15 carrier polymer is low density, linear low density, or ultra-low density
16 polyethylene.
- 17 13. The oxygen scavenging composition according to claim 7 wherein the
18 carrier resin is present in an amount in the range of from about 5 weight
19 percent to about 95 weight percent based on the total oxygen scavenging
20 composition.
- 21 14. The oxygen scavenging composition according to claim 10 wherein the
22 carrier resin is present in an amount in the range of from about 25 weight
23 percent to about 90 weight percent based on the total oxygen scavenging
24 composition.

- 1 15. The oxygen scavenging composition according to claim 11 wherein the
2 carrier resin is present in an amount in the range of from 50 weight percent
3 to 85 weight percent based on the total oxygen scavenging composition.
- 4 16. The oxygen scavenging composition according to claim 1, wherein the
5 catalyst is a transition metal salt.
- 6 17. The oxygen scavenging composition according to claim 16, wherein the
7 catalyst is a cobalt salt.
- 8 18. The oxygen scavenging composition according to claim 17, wherein the
9 catalyst is cobalt oleate, cobalt linoleate, cobalt neodecanoate, cobalt
10 stearate, or cobalt caprylate.
- 11 19. The oxygen scavenging composition according to claim 1 further
12 comprising a photoinitiator.
- 13 20. The oxygen scavenging composition according to claim 1 wherein the
14 carrier resin comprises an oxidizable polymer.
- 15 21. The oxygen scavenging composition according to claim 20 wherein the
16 oxidizable polymer is a substituted or unsubstituted ethylenically
17 unsaturated hydrocarbon polymer.
- 18 22. The oxygen scavenging composition according to claim 21 wherein the
19 oxidizable polymer is polybutadiene, polyisoprene, poly(styrene-butadiene),
20 poly(meta-xylenediamine-adipic acid), or polyacrylates which can be
21 prepared by transesterification of poly(ethylene-methyl acrylate) including
22 poly(ethylene-methyl acrylate-benzyl acrylate), poly(ethylene-methyl
23 acrylate-tetrahydrofurfuryl acrylate), poly(ethylene-methyl acrylate-nopol
24 acrylate), or mixtures thereof.

- 1 23. The oxygen scavenging composition according to claim 8, wherein the
2 polyterpene comprises a first phase and the catalyst comprises a second
3 phase, wherein the second phase is in sufficiently close proximity to the
4 first phase to catalyze an oxygen scavenging reaction.
- 5 24. The oxygen scavenging composition according to claim 23, wherein the
6 first phase forms a first layer and the second phase forms a second layer.
- 7 25. The oxygen scavenging composition according to claim 24, wherein the
8 second layer is in contact with the first layer.
- 9 26. The oxygen scavenging composition according to claim 24, further
10 comprising an oxygen barrier layer, a polymeric selective barrier layer, or a
11 heat seal layer.
- 12 27. An oxygen scavenging composition comprising at least one polyterpene, at
13 least one carrier resin, and at least one catalyst effective in catalyzing
14 oxygen scavenging, wherein the carrier resin exhibits a slower oxidation
15 rate than the polyterpene.
- 16 28. A film comprising the oxygen scavenging composition of claim 7.
- 17 29. An article comprising the oxygen scavenging composition of claim 7.
- 18 30. The article of claim 29 wherein the article is a package.
- 19 31. The article of claim 30 wherein the article is a package containing a food
20 product.
- 21 32. The article of claim 30 wherein the article is a package containing a
22 cosmetic, chemical, electronic device, pesticide or pharmaceutical.

- 1 33. The article of claim 29 wherein the article is a patch, bottle cap insert or
2 molded or thermoformed shape.
- 3 34. The article of claim 33 wherein the molded or thermoformed shape is a
4 bottle or tray.
- 5 35. A method for scavenging oxygen comprising placing an oxygen-sensitive
6 product in the package of claim 27.
- 7 36. A method for preparing an oxygen scavenging composition comprising melt
8 blending at least one polyterpene and at least one catalyst effective in
9 catalyzing oxygen scavenging.
- 10 37. The method of claim 36 further comprising melt blending at least one
11 carrier resin in the oxygen scavenging composition, wherein the carrier
12 resin exhibits a slower oxidation rate than the polyterpene.
- 13 38. The method of claim 37 further comprising at least one photoinitiator.

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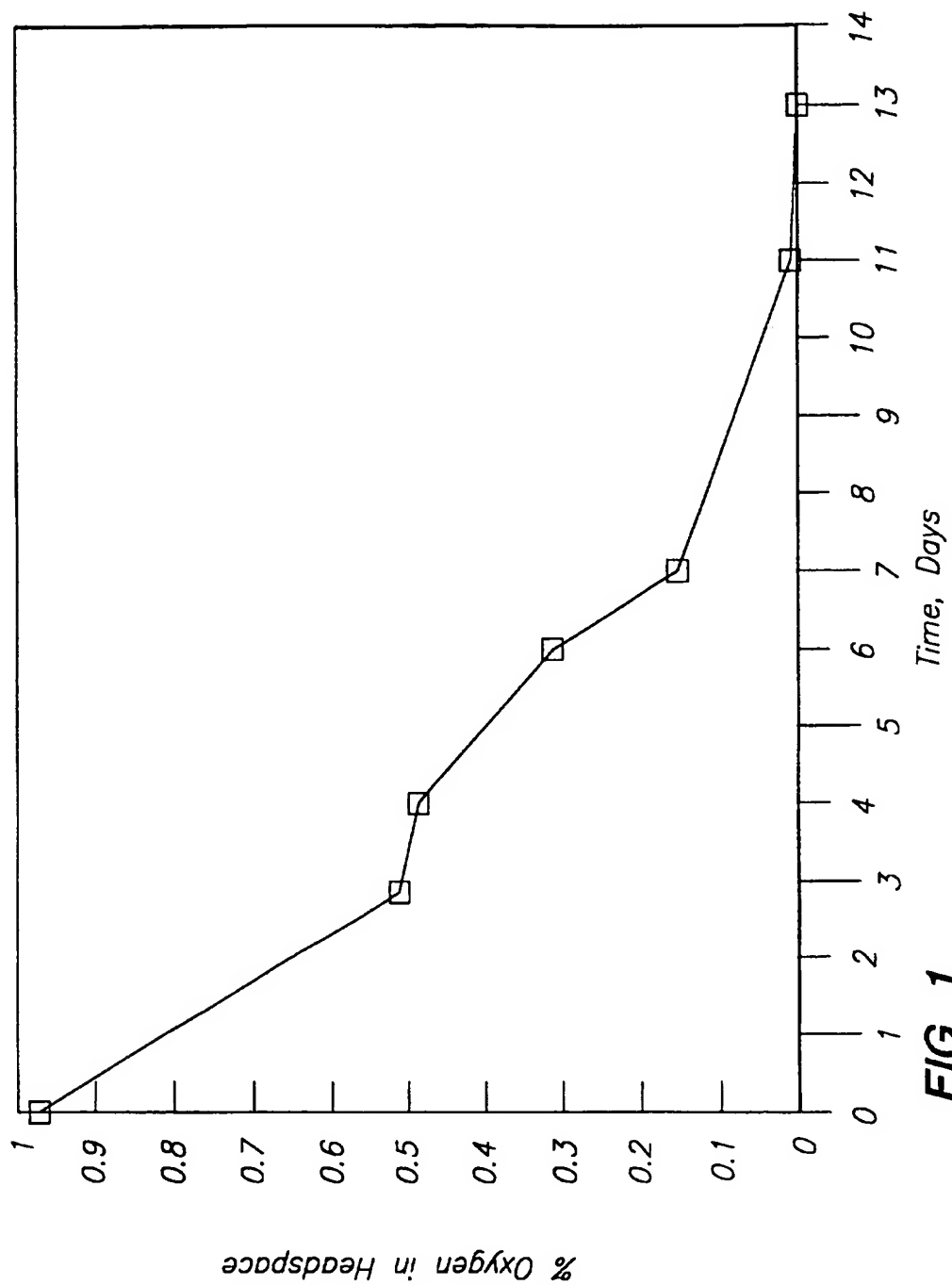


FIG. 1

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ALDEHYDES vs. Primary Oxidizable Component

Relative Concentration of Compound by GC-MS

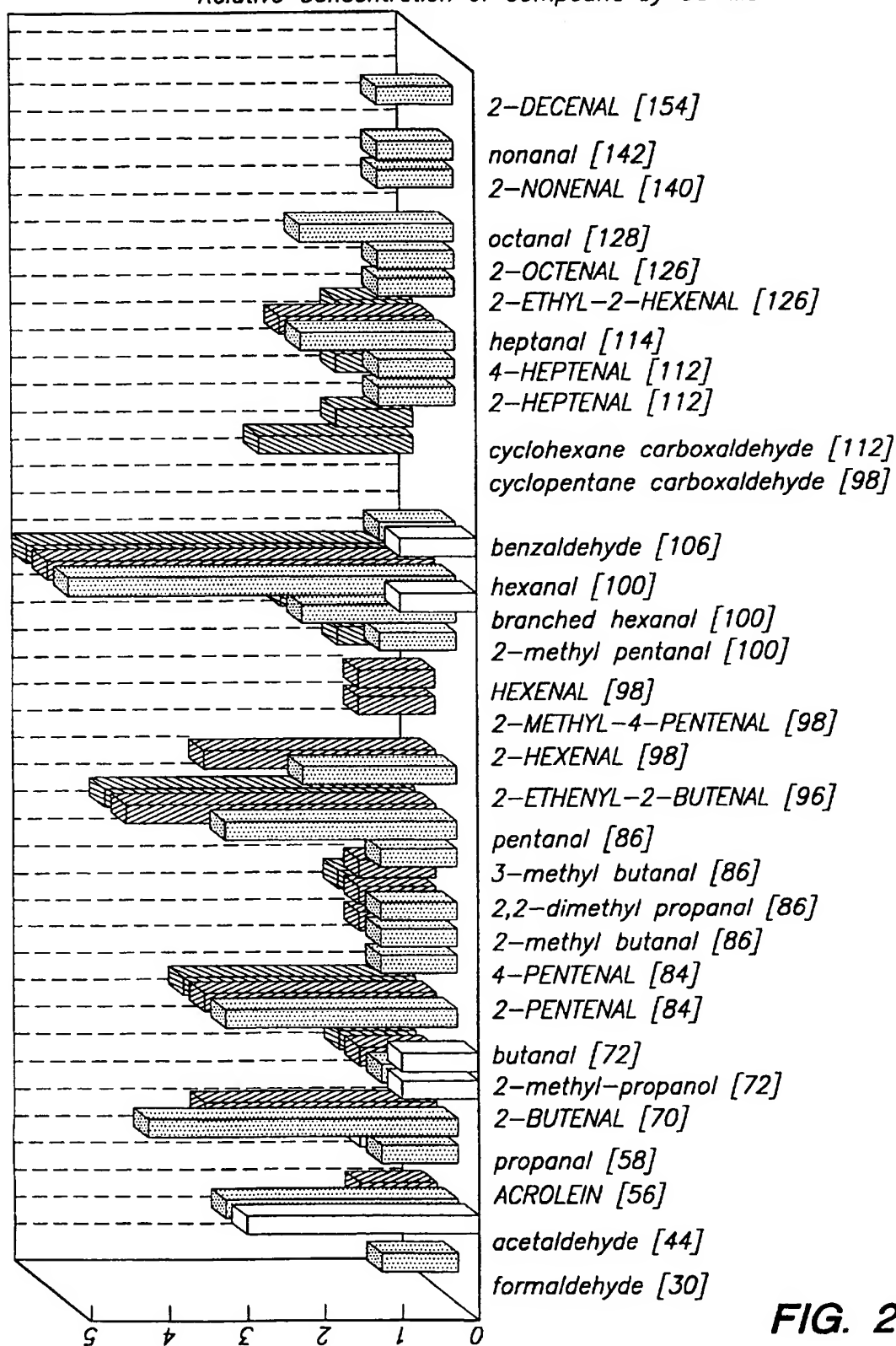
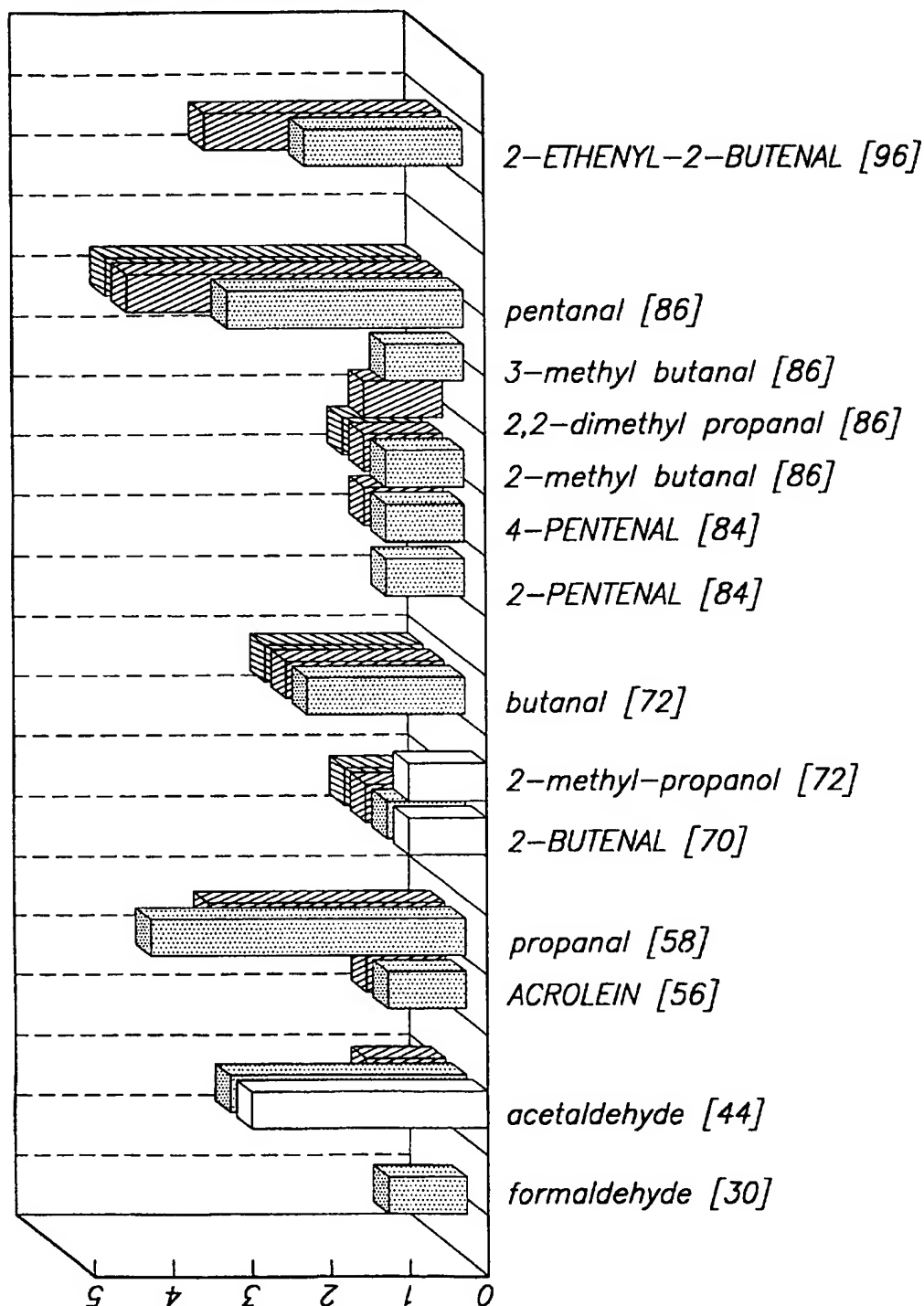


FIG. 2

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FIG. 3

ALDEHYDES vs. Primary Oxidizable Component
Relative Concentration of Compound by GC-MS



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ALDEHYDES vs. Primary Oxidizable Component

Relative Concentration of Compound by GC-MS

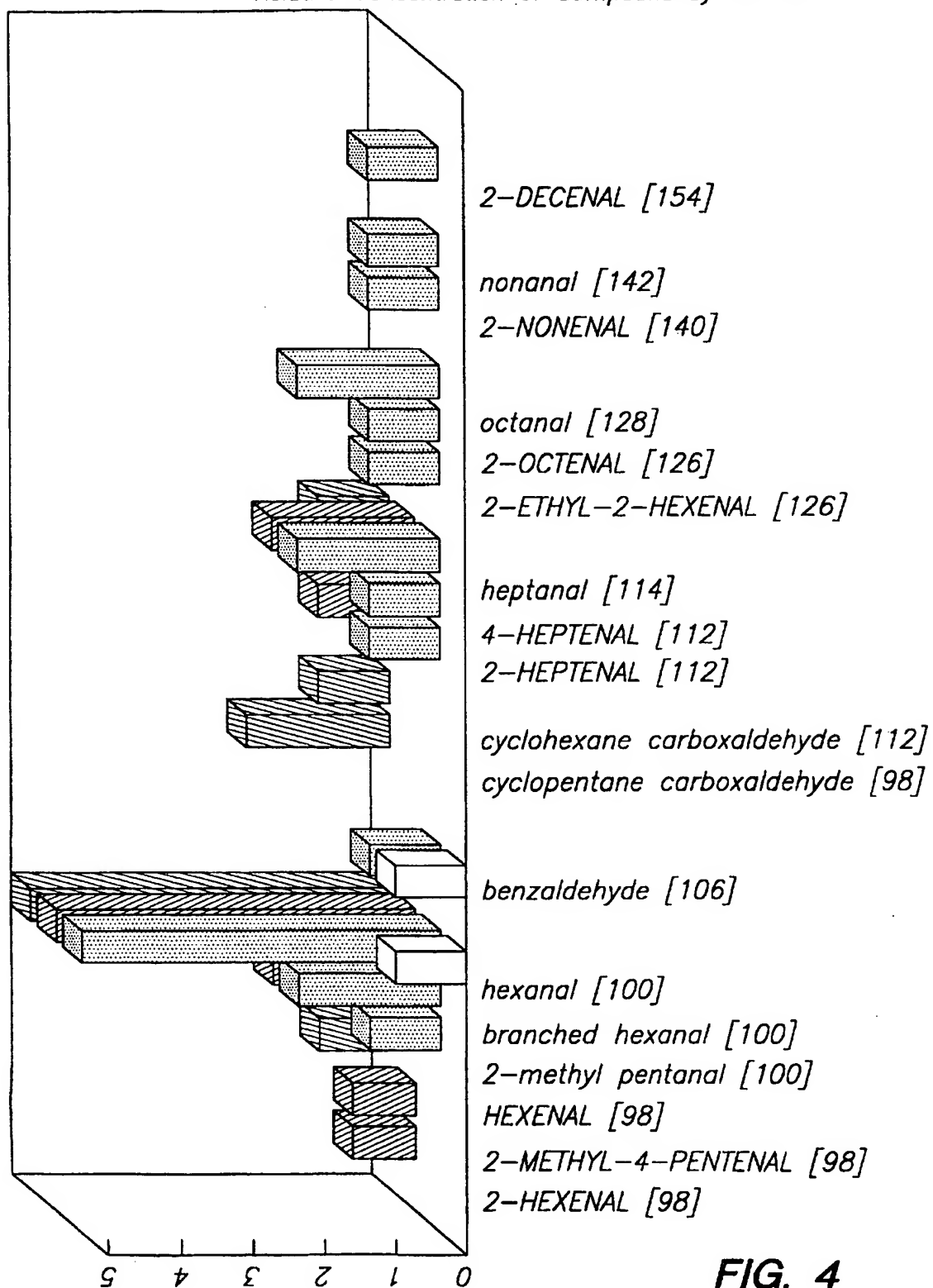
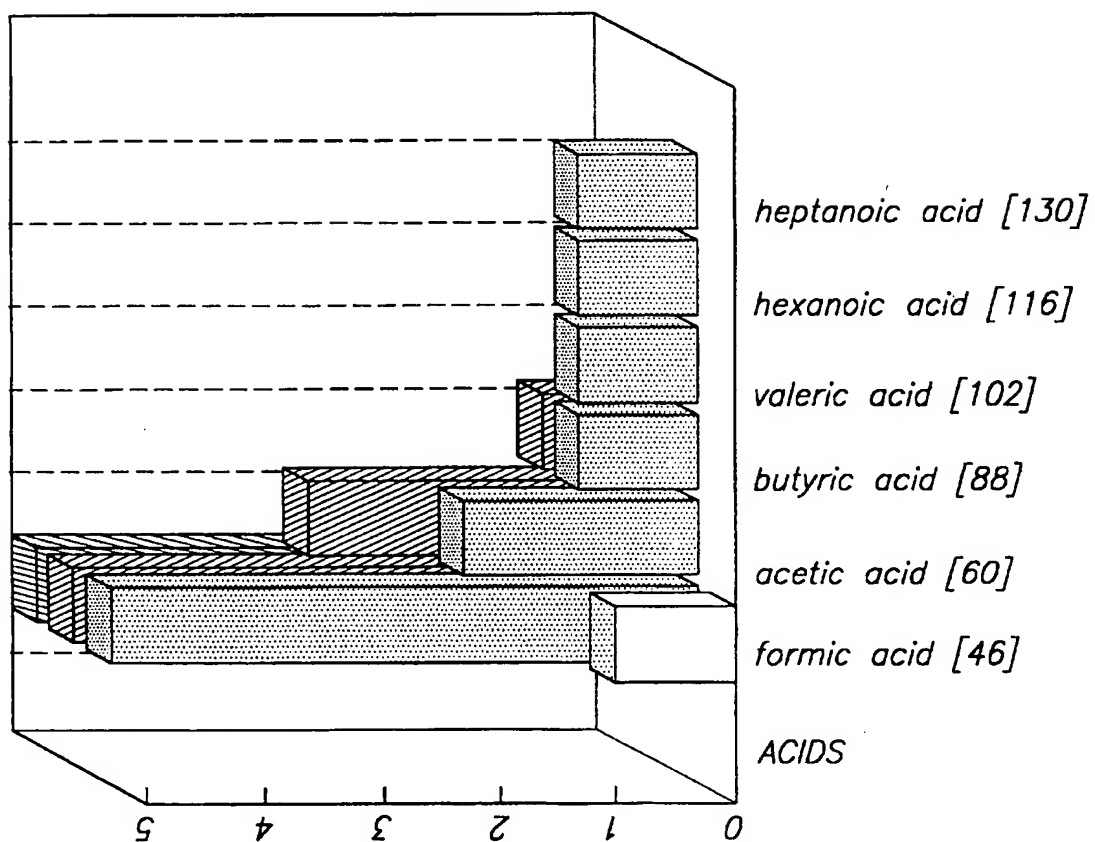
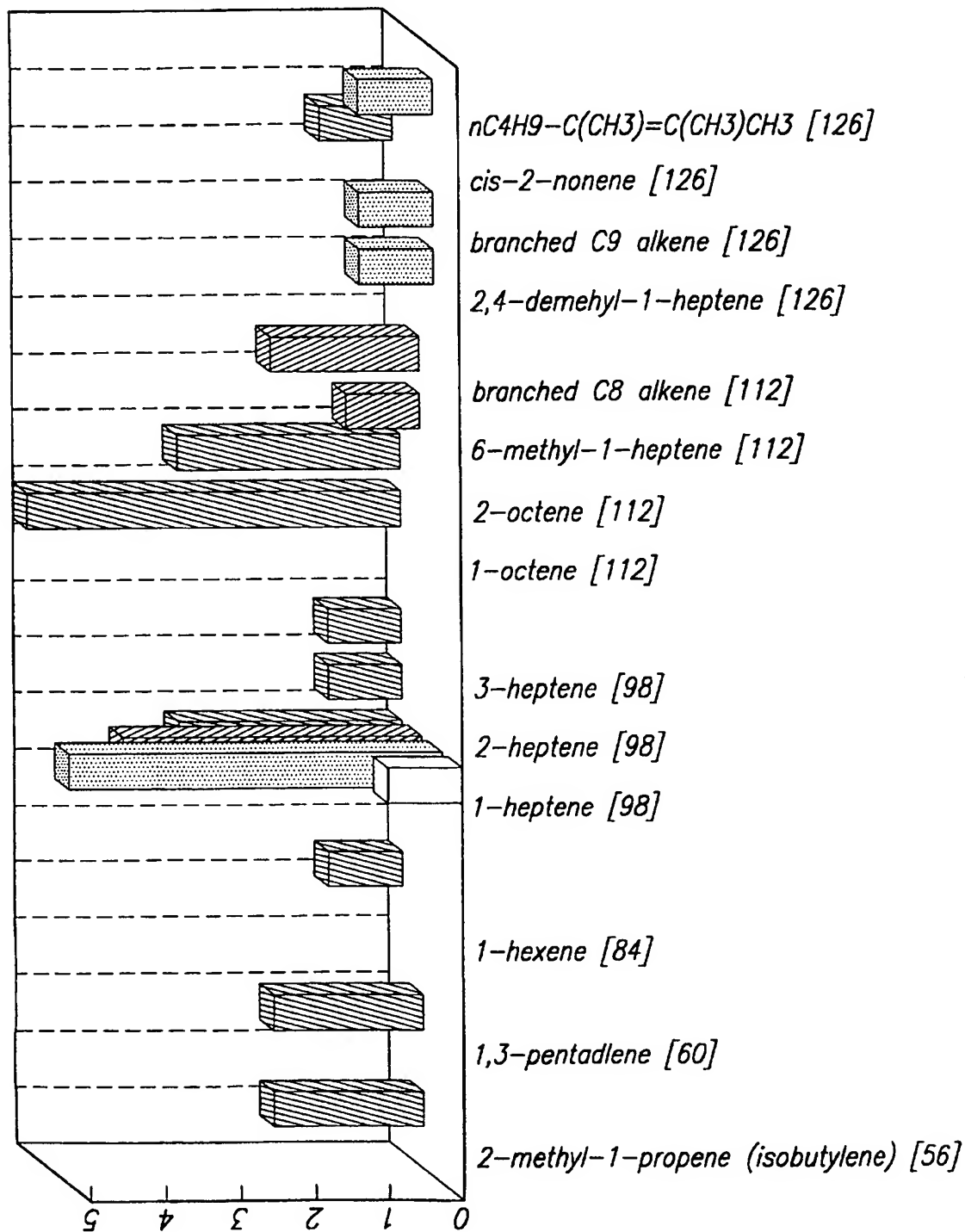


FIG. 4

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*ACIDS vs. Primary Oxidizable Component**Relative Concentration of Compound by GC-MS***FIG. 5**

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FIG. 6*ALKENES vs. Primary Oxidizable Component**Relative Concentration of Compound by GC-MS*

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 97/13015

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C08K5/098 C08L45/00 B32B27/08 A23L3/3436

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C08K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 96 25058 A (CHEVRON) 22 August 1996 cited in the application see claims 1,2,5-8,10,12-14,16-19 see claims 32,34-36; examples 3,4 ---	1,7,8; 23,29, 33-37
E	WO 97 32925 A (W.R. GRACE) 12 September 1997 see claims 1-4,17; table 1 ---	1,3,7,29
A	EP 0 507 207 A (W.R. GRACE) 7 October 1992	1,7, 16-18
X	see page 4, line 1 - line 6; claims 1,4,7,9,12,13,20,47; example 20 -----	1,16,18



Further documents are listed in the continuation of box C.



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